Table 1. Multidimensional NMR experiments

Experiment (acquisition of central peaks)*	Indirect† dimension(s)	t_{max}, ms; complex points	Measurement time, h	Relative sensitivity (peak pairs/ central peaks)
Sequential backbone connectivities (3D spectra)				
H$^{\alpha/\beta}$C$^{\alpha/\beta}$(CO)NHN	ω_1(^{13}C$^{\alpha/\beta}$/^{1}H$^{\alpha/\beta}$)	6.3; 95	9.2	(0.56/0.34)
(^{13}C)	ω_2(^{15}N)	21.5; 28		
HACA(CO)NHN	ω_1(^{13}C$^{\alpha}$/^{1}H$^{\alpha}$)	6.5; 54	5.4	(**1.00**‡/0.81)
(^{13}C)	ω_2(^{15}N)	21.5; 28		
Intraresidual backbone connectivities (3D spectra)				
HNNCAHA	ω_1(^{13}C$^{\alpha}$/^{1}H$^{\alpha}$)	6.6; 51	5.0	(0.41/0.27)
(INEPT)	ω_2(^{15}N)	21.5; 28		
H$^{\alpha/\beta}$C$^{\alpha/\beta}$COHA	ω_1(^{13}C$^{\alpha/\beta}$/^{1}H$^{\alpha/\beta}$)	6.3; 95	10.0	(0.22/0.11)
(^{13}C)	ω_2(^{13}C=O)	17.8; 32		
HNNCACB	ω_1(^{13}C$^{\alpha/\beta}$)	6.6; 56	8.0	(0.56)
	ω_2(^{15}N)	21.5; 28		
Intra- and sequential-backbone connectivities (3D spectrum)				
HNN(CO,CA)	ω_1(^{13}C$^{\alpha}$/^{13}C=O)	8.0/16.0§; 54	5.5	(0.54/1.41)
(INEPT)	ω_2(^{15}N)	21.5; 28		
Assignment of aliphatic resonances (3D spectra)				
HCCH-COSY	ω_1(^{13}C/^{1}H)	6.3; 95	6.2	(0.34/0.25)
(^{13}C)	ω_2(^{13}C)	6.4; 20		
HCCH-TOCSY¶	ω_1(^{13}C/^{1}H)	6.3; 95	7.0	(0.19/n.d.)
(^{13}C)	ω_2(^{13}C)	6.4; 20		

Assignment of aromatic resonances (2D spectra)				
HBCB(CGCD)HD (^{13}C)	ω_1(^{13}C/^{1}H)	6.3; 95	5.3	(0.45/0.33)
^{1}H-TOCSY-HCH-COSY[‖]	ω_1(^{13}C/^{1}H)	15; 150	3.4	(0.76/-)

One millimolar solution of "Z-domain" of *Staphylococcal* protein A at T = 25°C. The radio-frequency (rf) carrier for ^{1}H-frequency labeling in the projected "HC"-dimensions in which the chemical shifts of the aliphatic moieties are measured was set to 0 ppm. In 2D ^{1}H-TOCSY-HCH-COSY, the ^{1}H rf carrier was set to the position of the water line throughout. t_{max} denotes the maximal evolution time. The suite of experiments in this table can provide complete resonance assignments of proteins, excluding only the side chain NH_n moieties, the CH^{ε} groups of histidinyl, and the $CH^{\zeta 3}$, $CH^{\zeta 2,3}$, and $CH^{\eta 2}$ groups of tryptophanyl residues (which can be obtained as described in ref. 17). Notably, Z-domain does not contain tryptophans.

* Approach 1: Use of incomplete polarization transfer (rows labeled with "INEPT"); Approach 2: use of ^{13}C steady state magnetization (rows labeled with "^{13}C").

† Direct dimension: t_{max} = 73 ms/512 complex points.

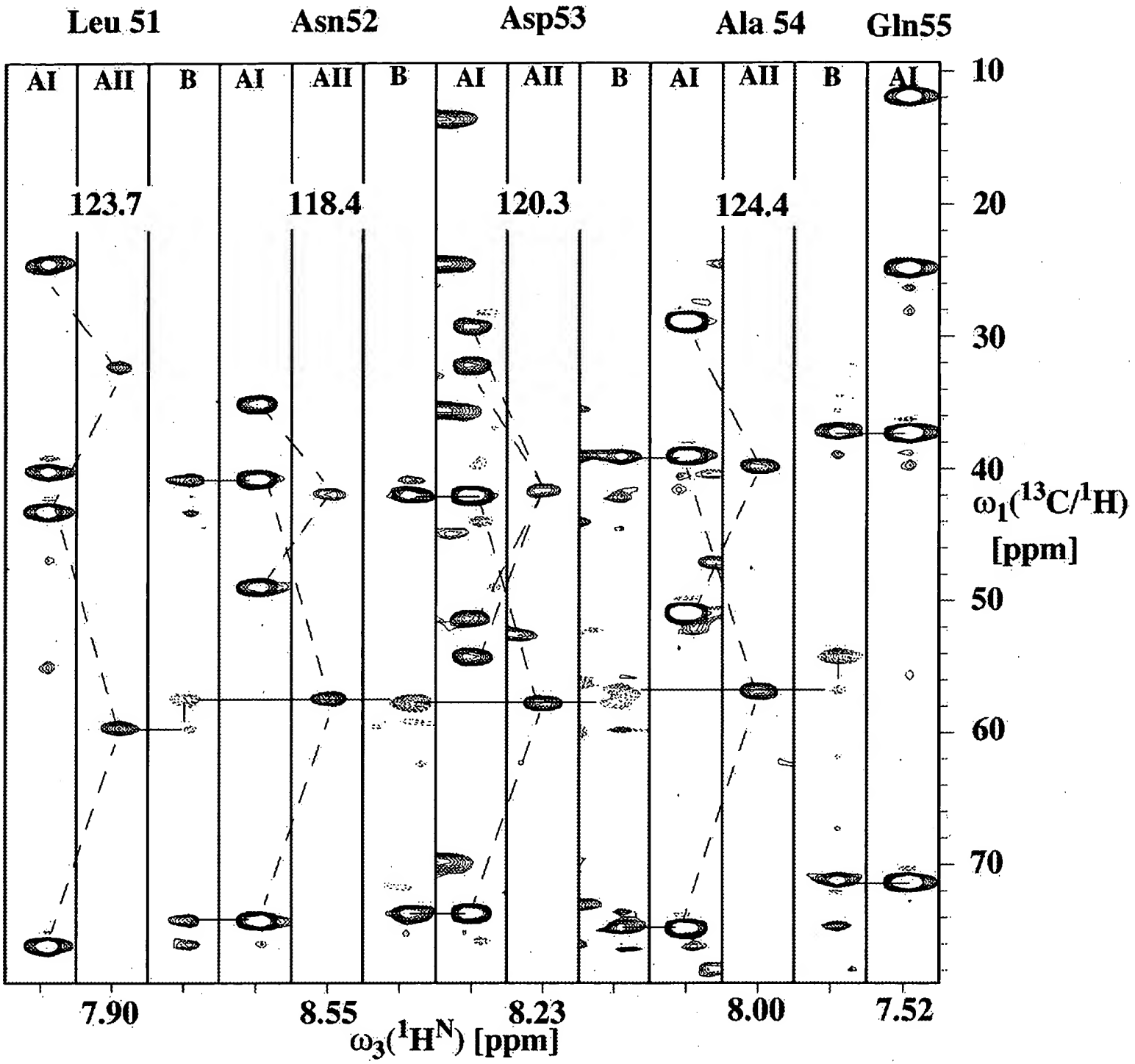
‡ The average signal-to-noise (S/N) ratio of peaks observed in this subspectrum was 33.2.

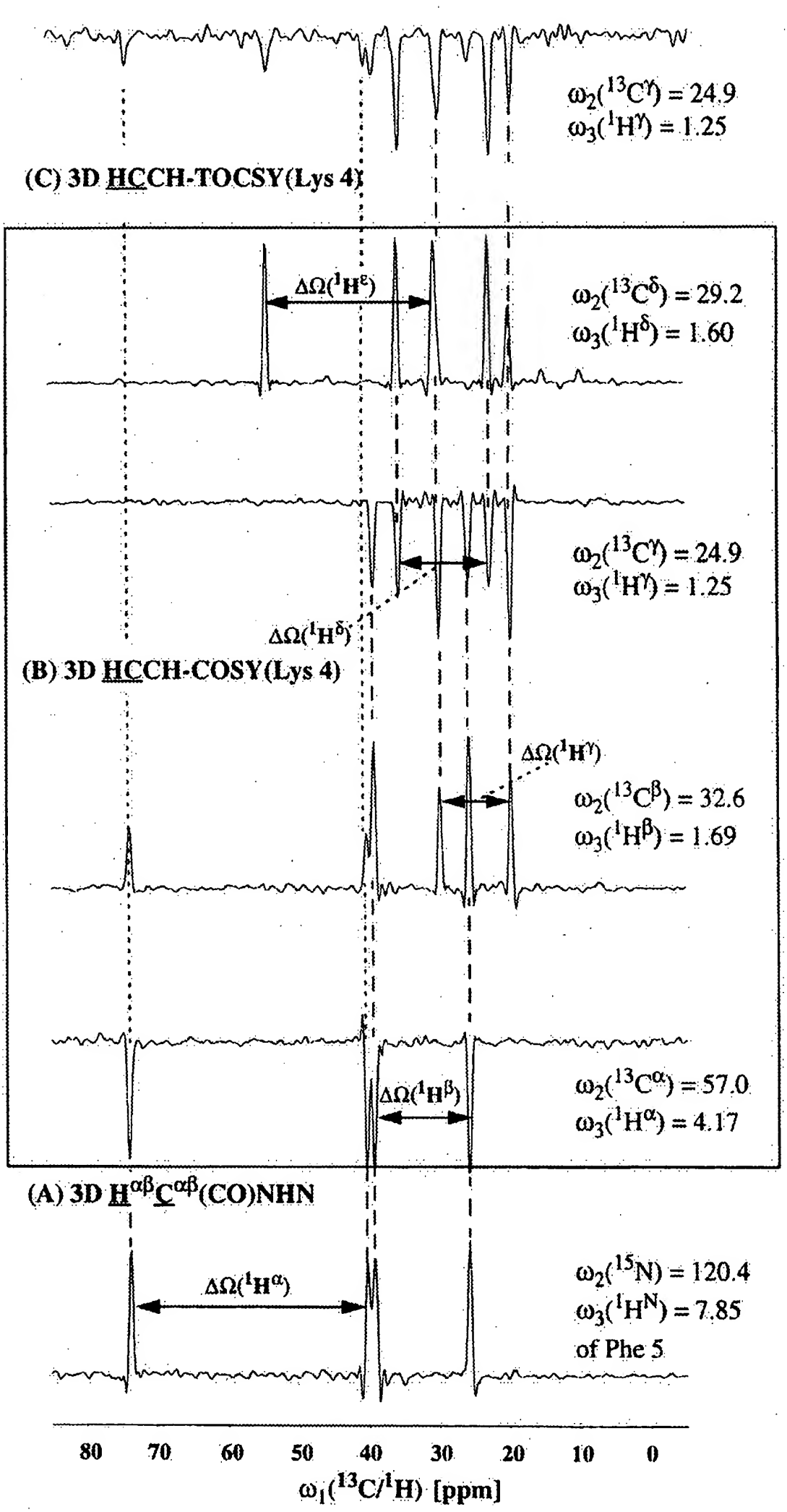
§ The increment for $^{13}C^{\alpha}$ chemical shift evolution was scaled by a factor of 0.5 relative to the values used to sample $^{13}C{=}{=}O$ evolution (5).

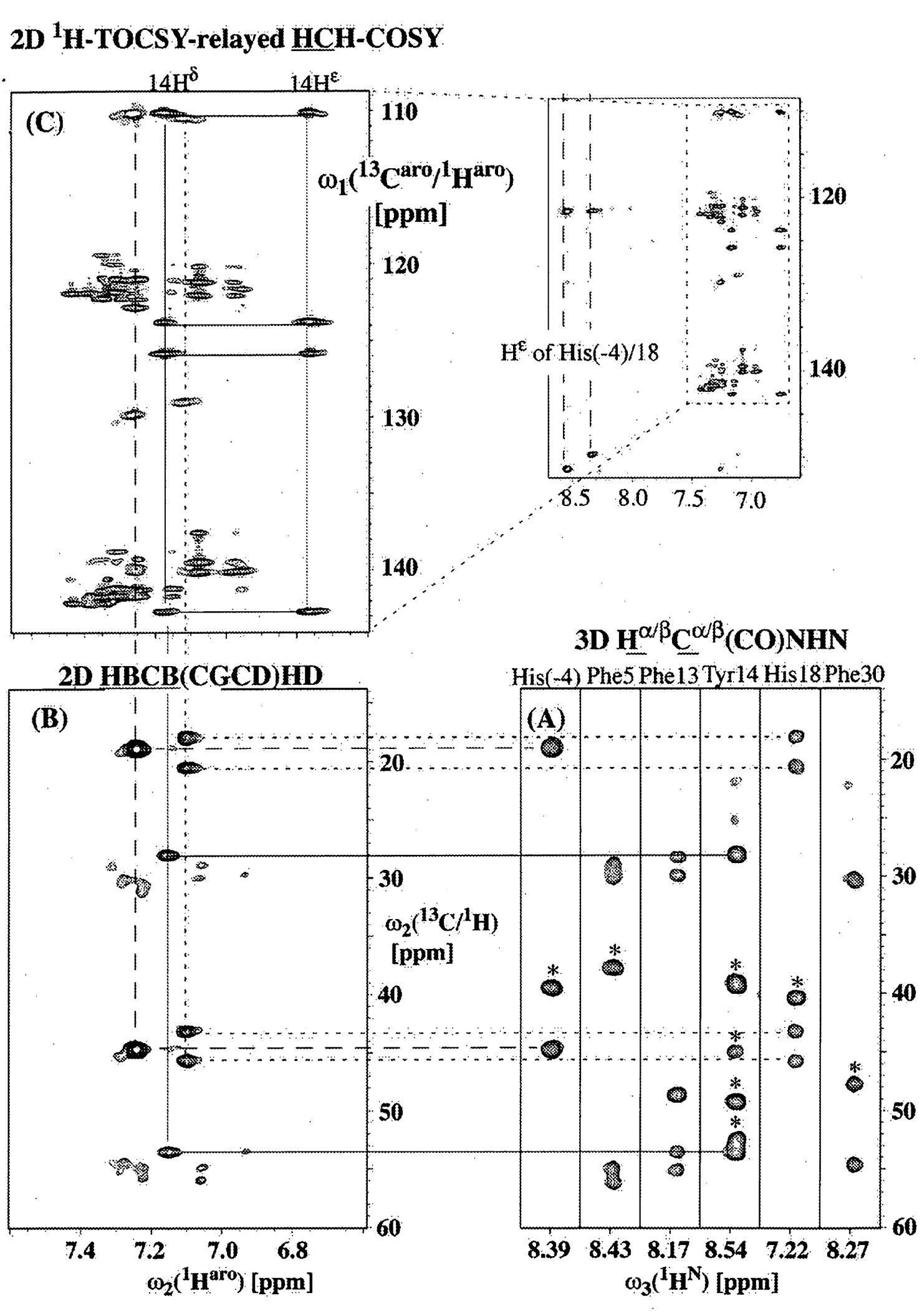
¶ The mixing times for the ^{13}C-TOCSY relay was set to 21 ms. The S/N ratios for the double-relay central peaks were too low to be accurately evaluated.

‖ The mixing time for the ^{1}H-TOCSY relay was set to 25 ms. The acquisition of central peaks is prevented by the use of spin-lock purge pulses (flanking the total correlation relay) to obtain pure phases.

[Return to Article]


Leu 51
Asn52
Asp53
Ala 54
Gln55
AI
AII
B
123.7
118.4
120.3
124.4
10
20
30
40
50
60
70
ω1(13C/1H) [ppm]
7.90
8.55
8.23
8.00
7.52
ω3(1HN) [ppm]


$\omega_2(^{13}C^{\gamma}) = 24.9$
$\omega_3(^1H^{\gamma}) = 1.25$
(C) 3D HCCH-TOCSY (Lys 4)
$\Delta\Omega(^1H^{\varepsilon})$
$\omega_2(^{13}C^{\delta}) = 29.2$
$\omega_3(^1H^{\delta}) = 1.60$
$\omega_2(^{13}C^{\gamma}) = 24.9$
$\omega_3(^1H^{\gamma}) = 1.25$
$\Delta\Omega(^1H^{\delta})$
(B) 3D HCCH-COSY (Lys 4)
$\Delta\Omega(^1H^{\gamma})$
$\omega_2(^{13}C^{\beta}) = 32.6$
$\omega_3(^1H^{\beta}) = 1.69$
$\Delta\Omega(^1H^{\beta})$
$\omega_2(^{13}C^{\alpha}) = 57.0$
$\omega_3(^1H^{\alpha}) = 4.17$
(A) 3D $H^{\alpha\beta}C^{\alpha\beta}$(CO)NHN
$\Delta\Omega(^1H^{\alpha})$
$\omega_2(^{15}N) = 120.4$
$\omega_3(^1H^N) = 7.85$
of Phe 5
80 70 60 50 40 30 20 10 0
$\omega_1(^{13}C/^1H)$ [ppm]


2D ^{1}H-TOCSY-relayed HCH-COSY
14H$^{\delta}$
14H$^{\varepsilon}$
(C)
110
120
130
140
ω_1(^{13}C^{aro}/^{1}H^{aro}) [ppm]
H$^{\varepsilon}$ of His(-4)/18
120
140
8.5
8.0
7.5
7.0
2D HBCB(CGCD)HD
3D H$^{\alpha/\beta}$C$^{\alpha/\beta}$(CO)NHN
His(-4) Phe5 Phe13 Tyr14 His18 Phe30
(B)
(A)
20
30
40
50
60
ω_2(^{13}C/^{1}H) [ppm]
7.4
7.2
7.0
6.8
ω_2(^{1}H^{aro}) [ppm]
8.39
8.43
8.17
8.54
7.22
8.27
ω_3(^{1}H^{N}) [ppm]